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PROVISIONAL SPECIFICATION

for the invention entitled:

"Fire Resistant Compositions"

The invention is described in the following statement:

FIRE RESISTANT COMPOSITIONS

The present invention relates to polymeric compositions which have useful fire resistant properties and which may be used in a variety of applications. The invention also relates to the preparation of such compositions and to their use. The present invention is illustrated with particular reference to electric cables, although it will be appreciated that the invention is more widely useful in the light of the associated benefits described herein.

Passive fire protection of structures and components is an area that is receiving increased attention. In this context the term "passive" means the use of materials that impart fire resistance. Passive fire protection systems are used extensively throughout the building and transportation industries and typically function by counteracting the movement of heat and/or smoke, by sealing holes, by prolonging stability of structures to which the system is applied and/or by creating thermal and/or physical barriers to the passage of fire, heat and smoke.

For many applications it is desirable that a material used to impart fire-resistance exhibits near net shape retention following exposure to the highest temperatures likely to be encountered in a fire situation (generally about 1000°C). If the material shrinks significantly, its integrity is likely to be compromised and it may also crack and/or fracture. In turn this can lead to a breakdown in thermal and electrical insulation and a loss of fire barrier properties and fire resistance. As will be apparent from the following, for many fire resistant polymeric compositions, their inherent shrinkage on exposure to elevated temperature is an accepted consequence of use. Specific measures taken to address this problem include the addition of intumescing agents, which cause expansion but provide a very mechanically weakened residue, or engineering design solutions which add to the cost of the final product or structure.

Electric cables applications typically consist of a central conductor surrounded by at least an insulating layer. Such cables find widespread use in buildings and indeed

form the basis for almost all electric circuits in domestic, office and industrial buildings. In some applications, e.g. in emergency power supply circuits, there is a requirement for cables that continue to operate and provide circuit integrity even when subjected to fire, and there is a wide range of standards for cables of this type. To meet some of these standards, cables are typically required to at least maintain electrical circuit integrity when heated to a specified temperature (e.g. 650, 750, 950, 1050°C) in a prescribed manner and for a specified time (e.g. 15 min., 30 min., 60 min., 2 hours). In some cases the cables are subjected to regular mechanical shocks during the heating stage. For example, they may be subjected to a water jet or spray either in the later stages of the heating cycle or after the heating stage. To meet a given standard a cable is typically required to maintain circuit integrity throughout the test. Thus it is important that the insulation maintains low conductivity (even after prolonged heating at high temperatures), maintains its shape so it does not shrink and crack, and is mechanically strong, particularly if it is required to remain in place during shock such as that resulting from mechanical impact due to water jet or spray exposure. It is also desirable that the insulation layer remaining after heating resists the ingress of water if the cable is required to continue operating during exposure to water spray for brief periods.

One method of improving the high temperature performance of an insulated cable has been to wrap the conductor of the cable with tape made with glass fibres and coated with mica. Such tapes are wrapped around the conductor during production and then at least one insulative layer is applied. Upon being exposed to increasing temperatures, the outer layer(s) are degraded and fall away, but the glass fibres hold the mica in place. These tapes have been found to be effective for maintaining circuit integrity in fires, but are quite expensive. Further, the process of wrapping the tape around the conductor is relatively slow compared with other cable production steps, and thus wrapping the tape slows overall production of the cable, again adding to the cost. A fire resistant coating that could be applied during the production of the cable by extrusion, thereby avoiding the use of tapes, would be desirable.

A variety of materials have been used to impart fire resistance to structures and components, including electric cables. The use of compositions based on silicone elastomers has been widespread. However, silicone elastomers can be expensive, have relatively poor mechanical properties and can be difficult to process, for example by extrusion techniques. Furthermore, these compositions tend to have the associated disadvantage that they are converted to powdery substances when exposed to fire because the organic components of the silicone elastomers are pyrolised or combusted. The pyrolysis or combustion products are volatilised and leave an inorganic residue or ash (silicon dioxide) that has little inherent strength. This residue is generally not coherent or self-supporting and indeed is often easily broken, dislodged or collapsed. This behaviour mitigates against using silicone elastomers as passive fire protection elements. This means, for instance, that silicone polymers used as insulation on electric cables must be protected and held in place with physical supports such as inorganic tapes and braids or metal jackets. On exposure to elevated temperatures, compositions in accordance with the present invention may form a physically strong coherent layer around an electrical conductor and therefore do away with the need to use such physical supports.

Certain compositions that exhibit fire-resistance do not also display suitably high electrical resistivity at elevated temperature. When used in cable applications these compositions provide only thermal insulation and/or a physical barrier between the conductor and supporting metal trays or brackets and tend to be electrically conducting in a fire situation leading to circuit failure. In this case additional steps must be taken to ensure electrical insulation is maintained at elevated temperature. For instance, a composition which imparts thermal resistance and/or provides a physical barrier at elevated temperature but which becomes electrically conducting may be provided over a separate layer specifically incorporated in the design to provide electrical insulation. It would be desirable to provide a single composition which confers the required thermal insulation and/or provides the required coherent physical barrier (eg no cracking or fracturing) at elevated temperature and which

furthermore functions as an electrical insulator at those temperatures. This is likely to provide significant cost savings and simplify product manufacture.

A further property often required of fire-resistant compositions is that they do not yield any potentially toxic gases or residues when exposed to a fire. Compositions of the present invention may also be inherently safe in this respect.

The present invention seeks to provide fire-resistant compositions which exhibit minimum and, preferably, no shrinkage when exposed to the kind of elevated temperatures associated with a fire. Furthermore, at such temperatures the compositions may also yield residue which is coherent and has good mechanical strength, even after cooling. The residue is self-supporting and will be retained in its intended position rather than fracturing and being displaced, for example, by mechanical shock. In this context the term "residue" is intended to describe the product formed when the composition is exposed to elevated temperature, as would be the case in a fire.

Desirably, as well as providing thermal insulation and/or a coherent physical barrier or coating, compositions in accordance with the present invention may also exhibit the required electrical insulating properties at elevated temperatures.

Compositions in accordance with the present invention may also have excellent processability enabling them to be manufactured and used with ease by conventional techniques. In addition the invention allows the preparation of fire resistant polymer products with a wide range of mechanical properties so that the invention can be tailored to suit the requirements of many different applications.

In general terms, the present invention provides a fire resistant composition which comprises inorganic components dispersed in a base composition comprising an organic polymer. The composition is converted into a solid ceramic material after exposure to elevated temperature. In this context a ceramic is an inorganic non-

metallic solid material prepared by high temperature processing (e.g. above about 400°C). The invention seeks to provide fire resistant compositions which undergo little or no change in dimensions when exposed to fire and which are capable of providing a residual coating that has coherence and adequate physical properties. Such compositions would have widespread application in providing fire resistance to structures and components thereof. The compositions are particularly useful for providing fire resistant insulation for electrical cables as they may provide suitably high electrical resistivity and breakdown strength, even after prolonged heating at high temperature. Use of a base composition comprising an organic polymer affords the potential for cost savings, enhanced processability and improved mechanical properties when compared with systems where the base composition is a silicone polymer.

Accordingly, in one embodiment, the present invention provides a fire resistant composition comprising:

at least 15% by weight, based on the total weight of the composition, of a base composition comprising at least 50% by weight of an organic polymer; and
at least 25% by weight, based on the total weight of the composition, of a silicate mineral filler,
wherein the fire resistant composition comprises a fluxing oxide in an amount of from 2 to 8% by weight of the residue obtained after heating the composition for 30 minutes at 1000°C.

It has been found that compositions in accordance with the present invention may form a coherent ceramic product when exposed to elevated temperatures and that this product exhibits desirable physical and mechanical properties. The ceramic char formed after exposure of compositions of the present invention at elevated temperature desirably has a flexural strength of at least 1 MPa. It is a distinct advantage that the compositions undergo little if any shrinkage following high temperature exposure. It is a further advantage, of the compositions of the present

invention, that this type of coherent product with desirable physical and mechanical properties can be formed at temperatures well below 1000°C. The compositions of the invention may be used in a variety of applications where it is desired to impart fire resistance to a structure or component. The compositions are therefore useful in passive fire protection systems.

The composition of the present invention includes as an essential component an organic polymer. Herein the term "organic polymer" embraces a variety of non-silicon containing polymers which are capable of forming a blend with the additional component(s) of the composition without any adverse incompatibility problems. Preferably, the organic polymer can accommodate high levels of inorganic additives, such as the silicate mineral filler, whilst retaining good processing and mechanical properties. It is desirable in accordance with the present invention to include in the fire resistant compositions high levels of inorganic filler as such compositions tend to suffer reduced weight loss on exposure to fire when compared with compositions having lower filler content. Compositions loaded with relatively high concentrations of silicate mineral filler are therefore less likely to shrink and crack when ceramified by the action of heat. The presence in the compositions of the invention of the specified range of fluxing oxide is also believed to contribute in this respect.

It is also advantageous for the chosen organic polymer not to flow or melt prior to its decomposition when exposed to the elevated temperatures encountered in a fire situation. Preferred polymers include ones that are cross-linked after the fire resistant composition has been formed, or ones that are thermoplastic but have high melting points and/or decompose to form a char near their melting points. Suitable organic polymers are commercially available or may be made by the application or adaptation of known techniques. Examples of suitable organic polymers that may be used are given below but it will be appreciated that the selection of a particular organic polymer will also be impacted by such things as the additional components to be included in the fire resistant composition, the way in which the composition is to be prepared and applied, and the intended use of the composition.

Useful thermoplastic polymers may be selected from homopolymers of olefins as well as copolymers of one or more olefins. Specific examples of suitable polymers include homopolymers of ethylene, propylene, butene-1, isobutylene, hexene, 1,4-methylpentene-1, pentene-1, octene-1, nonene-1 and decene-1. These polyolefins can be prepared using peroxide, Ziegler-Natta or metallocene catalysts, as is well known in the art. Copolymers of two or more of these olefins may also be employed. The olefins may also be copolymerised with other monomer species such as vinyl or diene compounds. Specific examples of copolymers which may be used include ethylene-based copolymers, such as ethylene-propylene copolymers and terpolymers (e.g. EPDM), ethylene-butene-1 copolymers, ethylene-hexene-1 copolymers, ethylene-octene-1 copolymers, ethylene-butene-1 copolymers and copolymers of ethylene with two or more of the above mentioned olefins.

The thermoplastic polyolefin may also be a blend of two or more of the above mentioned homopolymers or copolymers. For example, the blend can be a uniform mixture of one of the above systems with one or more of polypropylene, high pressure low density polyethylene, high density polyethylene, polybutene-1 and polar monomer-containing olefin copolymers such as ethylene/acrylic acid copolymers, ethylene/methyl acrylate copolymers, ethylene/ethyl acrylate copolymers, ethylene/butyl acrylate copolymers, ethylene/vinyl acetate copolymers, ethylene/acrylic acid/ethyl acrylate terpolymers and ethylene/acrylic acid/vinyl acetate terpolymers.

The organic polymer may include styrene monomers, such as polystyrene, SBR, SIS, SBS, SEBS.

The organic polymer may also be selected from poly(meth)acrylates, polyamides (including nylons), polyesters (such as PET), PVC, ABS, NBR, polyurethanes, epoxides, polycarbonates and polyoxymethylene acetals.

As noted, the organic polymer chosen will in part depend upon the intended use of the composition. For instance, in certain applications a degree of flexibility is required of the composition (such as in electrical cable coatings) and the organic polymer will need to be chosen accordingly based on its properties when loaded with additives. Polyethylenes and ethylene propylene elastomers, have been found to be particularly useful for compositions for cable coatings. Also in selecting the organic polymer account should be taken of any noxious or toxic gases which may be produced on decomposition of the polymer. The generation of such gases may be more tolerable in certain applications than others. Preferably, the organic polymer used is halogen-free.

The compositions of the present invention may also include a silicone polymer in combination with the organic polymer as the base composition in which the additional components are dispersed.

When used, the nature of the silicone polymer is not especially critical and one skilled in the art will be aware as to the type of polymers which may be used, although account should be had for the various issues described above in connection with the organic polymer (compatibility etc.). Useful silicone polymers are described in detail in the prior art including US 4,184,995, US 4,269, 753, US 4,269,757 and US 6,387,518. By way of more specific illustration, the silicone polymer may be an organopolysiloxane composed of units of formula:

$$R_r SiO_{\frac{4-r}{2}}$$

in which

R may be identical or different and are unsubstituted or substituted hydrocarbon radicals, r is 0, 1, 2 or 3 and has an average numerical value of from 1.9 to 2.1.

Examples of hydrocarbon radicals R are alkyl radicals, such as the methyl, ethyl, n-propyl, isopropyl, n-butyl, isobutyl, tert-butyl, n-pentyl, isopentyl, neopentyl, tert-pentyl and hexyl radicals, such as n-hexyl, heptyl radicals, such as the n-heptyl, octyl radicals, such as the n-octyl, and isooctyl radicals, such as the 2,2,4-trimethylpentyl,

nonyl radicals, such as the n-nonyl, decyl radicals, such as the n-decyl, dodecyl radicals, such as the n-dodecyl, octadecyl radicals, such as the n-octadecyl; cycloalkyl radicals, such as cyclopentyl, cyclohexyl and cycolheptyl and methyl cyclohexyl radicals; aryl radicals, such as the phenyl, biphenyl, napthyl and anthryl and phenanthryl; alkaryl radicals, such as o-, m- or p-tolyl radicals, xylyl and ethylphenyl radicals; and aralkyl radicals, such as benzyl and α- and β-phenylethyl.

Examples of substituted hydrocarbon radicals R are halogenated alkyl radicals, such as 3-chloropropyl, the 3,3,3-trifluoropropyl and the perfluorohexylethyl and halogenated aryl, such as the p-chlorophenyl and the p-chlorobenzyl.

The radicals R are preferably hydrogen atoms or hydrocarbon radicals having from 1 to 8 carbon atoms, preferably methyl. Other examples of radicals R are vinyl, allyl, methallyl, 1-propenyl, 1-butenyl and 1-pentenyl, and 5-hexenyl, butadienyl, hexadienyl, cyclopentenyl, cyclopentadienyl, cyclohexenyl, ethynyl, propargyl and 1-propynyl. The radicals R are preferably alkenyl radicals having from 2 to 8 carbon atoms, particularly vinyl.

The end groups of the polymers may be trialkylsiloxy groups, for example trimethylsiloxy or dimethylvinylsiloxy groups, or derived groups where one or more of the alkyl groups has been replaced by hydroxy or alkoxy groups.

The silicone polymer may be crosslinkable. The crosslinkable polymer can be any one which can be crosslinked by any one of the methods used for commercially available organopolysiloxane polymers including by free radical crosslinking with a peroxide through the formation of ethylenic bridges between chains, by addition reactions including reaction of silylhydride groups with allyl or vinyl groups attached to silicon, through condensation reactions including the reactions of silanols to yield Si-O-Si crosslinks, or using other reactive groups. Depending on the type of silicone polymer used the composition will therefore further comprise a suitable crosslinking agent. Suitable crosslinking agents are commercially available, for example there is a wide

range of useful peroxides suitable for use in this application, such as dibenzoyl peroxide, bis (2,4-dichlorobenzoyl) peroxide, dicumyl peroxide or 2,5-bis(tert-butylporoxy)-2,5-dimethylhexene or also mixtures of these, and when appropriate they may be included in the composition during the compounding process.

A silicone polymer type especially suitable for cable insulation is where the silicone polymer is of high molecular weight and has vinyl side chains that require heat to crosslink, either through platinum catalysed addition reactions or peroxide initiated free radical reactions. These silicone polymers are widely available commercially from major silicone producers.

The organopolysiloxane materials may also comprise reinforcing fillers such as precipitated or pyrogenic silicas and/or non-reinforcing fillers. Further, the surface of these silica type fillers may be modified by straight or branched organopolysiloxanes, organo-
chlorosilanes and/or hexamethyl disilazanes.

The organic polymer is present in the base composition in an amount of at least 50% by weight. This facilitates loading of the base composition with the additional components without detriment to the processability of the overall composition. As noted the based composition may include a silicone polymer. However, in this case the organic polymer would usually be present in the base composition in a significant excess when compared with the silicone polymer. Thus, in the base composition the weight ratio of organic polymer to silicone polymer may be from 5:1 to 2:1, for instance from 4:1 to 3:1. In terms of weight percentage, if present, the silicone polymer might generally be present in an amount of from 2 to 15% by weight based on the total weight of the formulated fire resistant composition. When a combination of organic and silicone polymers are used, high concentrations of silicone polymer can present processing problems and this should be taken into account when formulating compositions in accordance with the present invention.

The upper limit for the amount of base composition in the fire resistant composition tends to be influenced by the desired properties of the formulated composition. If the amount of the base composition exceeds about 40% by weight of the overall composition, it is unlikely that a cohesive, strong residue will be formed during a fire situation. Thus, the base composition generally forms from 15 to 40%, preferably from 20 to 30%, by weight of the formulated fire resistant composition.

The compositions in accordance with the present invention also include a silicate mineral filler as an essential component. Such fillers typically include alumino-silicates (e.g. kaolinite, montmorillonite, pyrophillite – commonly known as clays), alkali alumino-silicates (e.g. mica, felspar, spodumene, petalite) magnesium silicates (e.g. talc) and calcium silicates (e.g. wollastonite). Mixtures of two or more different silicate mineral fillers may be used. Such fillers are commercially available. Silicon dioxide (silica) is not a silicate mineral filler in the context of the present invention.

The silicate mineral filler may be surface treated with a silane coupling agent in order to enhance its compatibility with other materials present in the compositions of the present invention.

The compositions of the invention include at least 25% by weight, for instance at least 40% by weight and preferably at least 55% by weight, silicate mineral filler. The maximum amount of this component tends to be dictated by the processability of the composition. Very high levels of filler can make formation of a blended composition difficult. Usually, the maximum amount of silicate mineral filler would be about 70% by weight. The amount and type of silicate mineral filler used will also be dictated by the requirement to have a certain range of fluxing oxide in the residue formed by heating the composition at 1000°C for 30 minutes. As will be explained, the fluxing oxide can be generated *in situ* at elevated temperature by heating certain types of silicate mineral fillers (eg mica), to make the fluxing oxide become available at the surfaces of the filler particles. Additionally, or alternatively the fluxing oxide may come from a source other than the silicate mineral filler. As is explained later, the

fluxing oxide is believed to act as an "adhesive" assisting in formation of a coherent product at high temperature. The fluxing oxide is believed to contribute a binding flux at the edges of the filler particles. The presence of a high proportion of silicate mineral filler results in a composition which is likely to exhibit low shrinkage and cracking when a ceramic is formed at elevated temperature, and on cooling of the ceramic.

The compositions of the present invention also include a fluxing oxide as an essential component. By this is meant an oxide which forms a liquid flux at temperatures below about 1000°C. The generation of such a flux, as well as the amount generated, play an important role in yielding a ceramic structure having a desirable combination of properties following exposure at elevated temperature. As noted, the fluxing oxide may be generated by heating certain silicate mineral particles (eg mica) to make the fluxing oxide become available at the surface of the particles. Alternatively, or additionally, a fluxing oxide or precursor thereof may be added to the composition.

Without wishing to be bound by theory, it is believed that compositions in accordance with the present invention form a coherent ceramic product after exposure to elevated temperatures as a result of a fluxing oxide locally forming a eutectic composition at the interface of the silicate mineral filler particles and/or of other inorganic particles present in the composition or formed from decomposition thereof. These inorganic particles include other silicates minerals, and silicon dioxide (either added as an additional filler or generated by thermal decomposition of a silicon polymer or any silicone additive). When the fluxing oxide is added as a separate component to the composition, a eutectic forms at the interface between the fluxing oxide and contacting reactive particles. Ordinarily the silicate mineral filler, and any additional inorganic components, each have very high melting points. However, the presence of the fluxing oxide may result in eutectics at the interfaces of these causing melting at lower temperatures. The fluxing oxide causes formation of a eutectic which may act as a "bridge" between the particles of silicate mineral filler and other inorganic components present. This is thought to assist in "binding" the decomposition products

of the composition, silicate mineral filler, and, when present, other components. In this way formation of a coherent ceramic product is improved and it is possible to reduce the temperature required to form a comparatively strong porous ceramic material. It is very important to control the extent of eutectic formation and melting in the composition to control shrinkage and the creation of molten conductive pathways in the heated material. Compositions in accordance with the present invention may yield a coherent ceramic product that has essentially the same shape and volume as the composition before being exposed to elevated temperature. Such compositions can be described as retaining near net shape.

In general the fluxing oxide may be any compound which is capable of functioning in the manner described in order to form a ceramic product having the desired combination of properties. In practice, however, the fluxing oxide is likely to be boron oxide or a metal oxide selected from the oxides of lithium, potassium, sodium, iron, phosphorus, and barium. As mentioned, it is also possible to include in compositions of the present invention a precursor of the fluxing oxide (eg a silicate mineral such as mica), that is a compound that yields the fluxing oxide following exposure at the kind of elevated temperatures likely to be encountered in a fire. In that case the fluxing oxide is likely to be formed by thermal decomposition of the precursor. Similarly, when boric oxide is used as the fluxing oxide, it may be derived from a suitable precursor compound. Borates, and particularly zinc borate, provide useful precursors for boric oxide.

The fluxing oxide precursor may be a glass and a variety of glasses may be used. It should be noted however that to remain electrically insulating a low alkali metal content in the flux is desirable. The glass may take a variety of forms such as powder or fibres. Mixtures of one or more of these may be used. The preferred form is glass powder or frit. Irrespective of form, the glass additive preferably has a softening point below 1000°C, for example, below 800°C, and most preferably between 300 and 800°C. The softening point of the glass is defined by the temperature at which the viscosity of the glass equal $10^{7.6}$ poise. The glass additive may be one or a

combination of silicate, borate and/or phosphate glass systems. Suitable glass additives are commercially available.

As described, it is quite possible that one or more silicate mineral filler will contribute fluxing oxide following exposure at elevated temperature. In one embodiment, all of the fluxing oxides are derived from the silicate mineral filler(s). In another embodiment, the fluxing oxide is derived from the silicate mineral filler and another source, and this may lead to advantages in terms of the structure formed at elevated temperature due to fluxing oxide being provided from within particles of the silicate mineral filler and external to such particles. In a further embodiment the fluxing oxide is derived from the silicate mineral filler and an added boric oxide or a source of boric oxide (e.g. zinc borate). In a further embodiment the fluxing oxide is derived from the silicate mineral and added glass. In yet another embodiment fluxing oxide is derived from the silicate mineral added glass and boric oxide or a source of boric oxide. In a yet further embodiment the fluxing oxide is derived from a source or sources other than the silicate mineral filler.

In one embodiment the composition includes at least two different fluxing oxides which form liquid phases at different temperatures. This can enhance char integrity as well as ensuring that the composition functions as required over a broad temperature range.

The compositions should be formulated so that the residue formed after heating at 1000°C for 30 minutes contains 2% to 8% of a fluxing oxide regardless of the source of this oxide. In other words 8% by weight is the maximum amount of fluxing oxide that should be present in the residue obtained when the fire resistant composition of the invention is heated at 1000°C for 30 minutes. When the fluxing oxide is derived from a precursor, such as the silicate mineral filler or zinc borate or other additive, the amount of fluxing oxide may be calculated on the basis of the maximum amount of fluxing oxide this component would yield at elevated temperature. This calculation will, for instance, be based on the total amount of elements such as boron,

phosphorus, lithium, sodium, potassium, iron and barium which are present in the silicate mineral filler, borate and other additives and which can in theory result in formation of the corresponding fluxing oxides. To minimise shrinkage it is preferred that the amount of fluxing oxide is as low as necessary to enable formation of a coherent ceramic product on exposure to the kind of elevated temperature encountered in a fire. It has also been found that the physical form of the filler can influence shrinkage when the composition is heated. More specifically, it has been found that fillers composed of large platy particles provide reduced shrinkage.

Preferably, the compositions of the present invention include at least one silicate mineral that is an appreciable source of fluxing oxide. Mica satisfies this requirement and provides additional benefits because it is also available in plate form, making it a preferred component. The two most common classes of commercially available mica are muscovite and phlogopite, and these are therefore typically used in the present invention. Muscovite mica is a dioctahedral alkali aluminium silicate. Muscovite has a layered structure of aluminium silicate sheets weakly bonded together by layers of potassium ions. It has the following composition $KAl_3Si_3O_{10}(OH)_2$. Phlogopite mica is a trioctahedral alkali aluminium silicate. Phlogopite has a layered structure of magnesium aluminium silicate sheets weakly bonded together by layers of potassium ions. It has the following composition $KMg_3AlSi_3O_{10}(OH)_2$. Both mica types are typically present in the form of thin plates or flakes having sharply defined edges.

The composition of the present invention will invariably contain silicon dioxide as a result of being exposed to elevated temperature. For instance, this silicon dioxide may be derived from heating the silicate mineral filler. It may also come from thermal decomposition of a silicone polymer when included in the base composition. Silica may also be added as a separate filler component.

Other inorganic fillers used in polymer compounding can be included in the compositions, these include aluminium oxide, aluminium hydroxide, magnesium hydroxide, magnesia, dolomite, zinc oxide and zirconia. Also inorganic fibres which

do not melt at 1000°C can be incorporated, including aluminosilicate fibres. This may lead to a reduction in shrinkage at elevated temperature.

Usually, after exposure at elevated temperature (to 1000°C) the residue remaining will generally constitute at least 50%, preferably at least 55% and more preferably at least 60%, by weight of the composition before pyrolysis. Higher amounts of residue are preferred as this may improve the char (ceramic) strength at all temperatures by better mechanical interlocking of particles and also a reduced tendency to shrink.

As mentioned, it has also been found that the mechanical properties of the ceramic formed from the composition of the present invention can be enhanced by including in the composition a low level of boric oxide or precursor thereof which yields boric oxide at elevated temperature e.g. zinc borate. In this case however the total amount of boric oxide and other fluxing oxides will not exceed 8% by weight of the residue obtained after heating the composition for 30 minutes at 1000°C.

It has also been found that removing volatile decomposition products from fillers such as clay by calcining prior to addition to the composition reduces shrinkage when the composition of the invention is heated at high temperature. This is believed to help reduce mass loss and volume shrinkage of the composition when exposed to elevated temperature.

As explained, preferably the compositions exhibit minimal shrinkage after exposure to the kind of temperatures likely to be encountered in a fire. By this is meant that the maximum volume change in a product formed from a composition in accordance with the present invention is usually about 5% and preferably about 2%. Most preferably, the composition is formulated to give a net shape reduction of no more than 1%, and desirably less than this.

Compositions in accordance with the invention may also exhibit the electrical insulating properties at high temperature that are required for use in electric cables.

Essentially this means that the electrical resistance of the material, while less than at room temperature, does not fall to a point where the normal operating voltage can overcome the insulation resistance of the material and cause a short circuit.

The compositions of the invention are also preferably free of other elements that may constitute a health and safety problem due to toxicity. Thus, the compositions are preferably free of halogen compounds. For the same reason, while lead oxide and antimony oxide could be used as fluxing oxides, usually the compositions of the present invention are free of lead and antimony.

For cable applications, where the electrical resistivity of the composition is important, the levels of alkali ions present must be carefully considered as they can cause electrical conductivity at high temperatures. For example in a given composition, if the level of mica is too high electrical integrity problems arise due to an unacceptable reduction in electrical resistivity of the composition and/or from dielectric breakdown when the compositions are subjected to high temperatures for an extended period of time. At high temperatures alkali metal ions, for instance from mica, tend to provide conductive pathways, resulting in the need to limit the level of mica.

In one embodiment of the present invention it has been found that having a relatively high concentration of fluxing oxides in the composition of the invention can lead to formation of a glassy surface layer when the composition is ceramified (at elevated temperature) and cooled. Desirably, this surface layer has been found to confer improved water resistance to the ceramic formed. The formation of such a surface layer, and associated enhanced water resistance, is particularly beneficial in electrical cable applications because ingress of water (used to quench a fire) through the ceramic is likely to lead to electrical shorting. Of course, the potentially detrimental effects of high levels of a glass phase (shrinkage and electrical conductivity) must be taken into account. The amount of fluxing oxide required to form the glassy surface layer when the composition forms a ceramic may vary depending upon the layer thickness (see below) and other ingredients present in the composition. However, in

general terms the fluxing oxide level is desirably more than 5% of the residue obtained after heating the composition for 30 minutes at 1000°C. The total amount of glass phase present in the heated composition may be derived from a single source or from more than one source. For instance, the glass phase may be derived predominantly from glass frits, fibres and/or particles of the same or different type glass. A similar effect may be observed by using a relatively high concentration of mica, for example about 25% by weight, since this too can lead to the formation of sufficient liquid phase during heating.

The mechanism by which the glassy surface layer (skin) is formed is not clearly understood, although glass flow is clearly required in order to form the (densified) glassy surface layer. This means that the melting temperature of the glass additive and/or the liquid phase formed by fluxing oxides from other sources must be selected so that some flow is possible at the ceramic-forming temperature. It may be desirable to incorporate a variety of glass phases with different melting points to achieve skin formation and the desirable mechanical properties. The mechanism for formation of the glassy surface layer may be associated with surface tension effects between the molten glass and its local environment. One possible explanation for migration and aggregation of glass to the surface of the formed ceramic is that the surface energy at the glass/atmosphere interface is lower than that of the energy at the interface between the molten glass and the bulk of the composition. This being so, the molten glass migrates to the lower energy interface.

It has been found that the thickness of the composition may have an impact on the formation of the water resistant surface layer. This is believed to be due to volume effects, with more glass (and/or mica) being available for formation of a suitably thick surface layer when the thickness of the composition is greater. It has been observed in fact that a thicker sample of a composition yields a more water resistant surface layer than a thinner sample of the same composition.

Water resistance can also be improved by the addition of inorganic fibres which do not melt at 1000°C. Alumino-silicate fibres are preferred and can be used at levels of up to 5% by weight.

Other components may be incorporated into the compositions of the present invention. These other components include lubricants, plasticisers, inert fillers, antioxidants fire retardant materials, fibre reinforcing materials, materials that reduce thermal conductivity (eg exfoliated vermiculite), chemical foaming agents (which serve to reduce density, improve thermal characteristics and further enhance noise attenuation), and intumescing materials (to obtain a composition that expands upon exposure to fire or elevated temperature). Suitable intumescing materials include natural graphite, unexpanded vermiculite or unexpanded perlite. Other types of intumescing precursors may also be used. The total amount of such additional components does not usually exceed 20% by weight based on the total weight of the composition.

The compositions of the present invention may be prepared by blending and heating the various components. Heating may be required for peroxide crosslinking of the polymer component(s). Any conventional compounding equipment may be used. If the composition has relatively low viscosity, it may be processed using dispersing equipment, for instance of the type used in the paint industry. Materials useful for cable insulation applications are of higher viscosity (higher molecular weight) and may be processed using a two roll mill, internal mixers, twin-screw extruders and the like. Depending upon the type of crosslinking agent/catalyst added, the composition can be cured by exposure to air at 200°C, in an autoclave with high pressure steam, using continuous vulcanisation equipment including a liquid salt bath and, conceivably, by exposure to any medium that will cause the peroxide to decompose, including microwaves, ultrasonics etc.

The compositions of the present invention may be used in a large number of applications where fire resistance is desired. For example, the compositions may be

used to form a fire resistant building panel or in the manufacture of ceramic products. The composition may be used by itself or together with one or more layers of other materials.

The compositions of the present invention may be provided in a variety of different forms, including:

1. As a sheet, profile or complex shape. The composition may be fabricated into these products using standard polymer processing operations, eg extrusion, moulding (including hot pressing and injection moulding). The products formed can be used in passive fire protection systems. The composition can be used in its own right, or as a laminate or composite with another material (for example, plywood, vermiculite board or other). In one application the composition may be extruded into shapes to make seals for fire doors. In the event of a fire, the composition is converted into a ceramic thus forming an effective mechanical seal against the spread of fire and smoke.

2. As a pre-expanded sheet or profile. This form has additional benefits compared with the above, including reduced weight and the capacity for greater noise attenuation and insulation during normal operating conditions. Porosity can be incorporated into the material during manufacture of the sheet or profile by thermal degradation of a chemical blowing agent to produce a gas product, or by physically injecting gas into the composition before curing.

3. As an intumescent product, which expands by foaming when exposed to heat or fire. In this application the product can be used, for example, around pipework or penetrations between walls. In the event of a fire the product expands to fill the void and provide an effective plug to prevent the spread of fire. The intumescent material may be in the form of an extrudable paste or a flexible seal.

4. As a mastic material which can be applied (for example from a tube as per a conventional silicone sealant) as a seal for windows and other articles.

5. As a paint, or an aerosol based material, that could be sprayed or applied by with a brush.

The compositions of the present invention may also be used in the manufacture of ceramics. In this aspect, the present invention provides a method for manufacturing a ceramic article comprising a composition as herein described. The method involves shaping said composition to a desired shape and heating to elevated temperatures to thereby degrade the polymers and form a solid material such that a coherent mass is formed after cooling. Other conventional ceramic processing steps, such as sintering, may also be included in this method. The present invention also provides a method for producing ceramics using polymer precursors involving organic polymers, or blends of organic polymers with silicone and other organosilicon polymers. In this aspect of the present invention, it has been found that the heating step causes little or no volume reduction in the article. Thus, the firing step can result in the production of near net shapes.

Specific examples of passive fire protection applications where this invention may be applied include but are not limited to firewall linings for ferries, trains and other vehicles, fire partitions, screens, ceilings and linings, structural fire protection [to insulate the structural metal frame of a building to allow it to maintain its required load bearing strength (or limit the core temperature) for a fixed period of time], fire door inserts, window and door seals, intumescent seals, and compounds for use in electrical boxes, in fittings, straps, trays etc that are attached to or used to house cables or similar applications.

The compositions of the present invention are especially useful in the coating of conductors. The compositions are therefore suitable for the manufacture of electrical cables that can provide circuit integrity in the case of fire. In the design of such

cables the compositions can be used as an extruded insulation directly over conductors, as an interstice filler in multi-core cables, as individual extruded fillers added to an assembly to round off the assembly, as an inner layer prior to the application of wire or tape armour or as an outer sheathing layer.

In practice the composition will be extruded onto the surface of a conductor. This extrusion may be carried out in a conventional manner using conventional equipment. Typically the composition will be crosslinked immediately after extrusion. The thickness of the layer of insulation will depend upon the requirements of the particular standard for the size of conductor and operating voltage. Typically the insulation with have a thickness from 0.6 to 3 mm. For example, for a 35 mm^2 conductor rated at 0.6/1 kV to Australian Standards would require an insulation thickness of approximately 1.2 mm. As noted, compositions in accordance with the invention may exhibit excellent thermal and electrical insulating properties at elevated temperature. When used such compositions enable a cable of elegantly simple design to be manufactured since there is then no need to include a distinct layer to confer electrical insulating properties. According to this aspect the present invention provides electrical cables consisting of a suitable composition in accordance with the present invention provided directly on a conductor. The cable may include other layers such as a cut-resistant layer and/or sheathing layer. However, the cable does not require an additional layer intended to maintain electrical insulation at elevated temperature.

Examples

A two-roll mill was used to prepare the compositions described below. The ethylene propylene rubber (and any silicone polymer) were banded on the mill (10-20°C) and other components were added and allowed to disperse by separating and recombining the band of material just before it passed through the nip of the two rolls. When these were uniformly dispersed, the peroxide was added and dispersed in a similar manner.

The compositions tested were fabricated into the required shapes and cross-linked by

heating for 30 minutes at 170°C and 7 MPa.

The fluxing oxide weight contributed to the residue after 100g of each of the clay, talc and mica used in the examples was heated at 1000°C for 30 minutes was 1.0g, 1.7g and 11.1g respectively. The residue content of each was 86.1g, 96.0g, and 96.9g respectively.

Example 1

A number of compositions (see Table 1) were prepared and are denoted A-T. A rectangular sheet of each composition was made with dimensions 30 mm x 13 mm x 2 mm (approx). The samples were fired under slow firing conditions (heating from room temperature to 1000°C at a temperature increase rate of 12°C/min followed by holding at 1000°C for 30 minutes) or fast firing conditions (putting sheets into a pre-heated furnace at 1000°C and maintaining at that temperature for 30 minutes). After firing, each sample took the form of a ceramic char, the dimensions of which were measured in order to determine the linear shrinkage resulting from firing. The flexural strength of cooled samples was determined in a three point bend test using a universal testing machine.

Composition A is an example of a basic composition that consists of only one organic polymer, silicate mineral fillers, a small amount of a fluxing oxide and some additives.

Sample B is a composition comprising a blend of an organic polymer with a small amount of a silicone polymer which is a source of silica for char formation. This composition does not contain any separately added fluxing oxides (all the fluxes are derived from mineral fillers).

Sample C uses a composition that contains a small amount of glass frit as a source of fluxing oxide. Comparison of Samples B and C shows that the addition of a small amount of a glass as a source of fluxing oxide can improve char strength.

Comparison of Samples C and D shows that some silicate mineral fillers, in this case clay, can lead to much higher char shrinkage than other fillers.

Comparison of Samples D and E shows that adding higher amounts of glass frit results in increased shrinkage and char strength.

Comparison of Samples F and G shows that removing volatile decomposition products from fillers such as clay by pre-calcining them reduces the char shrinkage with no significant adverse effect on char strength.

Comparison of Samples G and H shows that more talc and less clay is favourable for reducing the char shrinkage.

Comparison of Samples A and H shows that the effect of boric oxide is independent of the type of source used (zinc borate or boric oxide) provided the quantity of boric oxide is the same. This also shows that zinc oxide introduced by zinc borate has no noticeable role in char shrinkage or strength. Its effect is similar to that of refractory oxide alumina.

Comparison of Samples J and K shows that higher amounts of boric oxide results in higher amounts of shrinkage.

M Sample contains aluminium hydroxide and silicate mineral fillers with no separately added fluxing oxide.

N is an example of a composition that does not contain any clay or talc, but contains aluminium hydroxide, mica and wollastonite.

Comparison of Samples O and P shows that larger particles of a mineral filler reduce shrinkage.

Comparison of Samples Q, R and S shows that addition of fine silica either as silica powder or as a silicone polymer that decomposes giving silica powder causes an increase in shrinkage and strength of char.

Table 1

	A	B	C	D	E	F	G	H	J	K	L	M	N	O	P	Q	R	S	T
Ethylene propylene rubber	22	22.4	22	22	22	22	22	22	22	22	22	20	22	22	22	22	22	23.4	22
Silicone rubber		5.8	6	6	6							4.8	6	6	6	1	5		6
Clay	10			24	21		30	10	10	10		19.2		14	14	24	24	25.5	24
Calcined clay						30.0													
Talc	44	31.0	28	14	14	23	23	43	52	49	64·	12		14	14	14	14	14.9	14
Muscovite Mica	9	29.1	30	20	20	9	9	9				16	20	20	20	20	20	21.3	20
Zinc Borate						4	4	4	4	7	2								
Glass Frit (flux content - 5.1%)			2	2	5								2	2	2	2	2	2.1	2
Fine silica																5	1		
Boric Oxide	1.35																		
Alumina	1.65																		
Coarse Wollastonite														10					
Fine Wollastonite													18		10				
Aluminium hydroxide												20	20						
Peroxide	3	2.3	3	3	3	3	3	3	3	3	3	2.4	3	3	3	3	3	3.2	3
Other additives (lubricants, plasticisers, antioxidents etc)	9	9.4	9	9	9	9	9	9	9	9	9	5.6	9	9	9	9	9	9.6	9
Total	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Firing Condition	Slow	Slow	Slow	Fast	Fast	Fast	Fast	Slow	Slow	Slow	Slow	Fast	Fast	Slow	Slow	Fast	Fast	Fast	Fast
Shrinkage of char - %	3.8	0.5	1.2	6.1	8.8	5.4	7.0	3.4	3.3	6.3	2.0	3.1	0.0	3.9	4.8	6.0	5.7	3.2	6.8
Flexural strength of char - MPa	8.2	1.1	2.6	3.1	9.4	5.2	5.3	7.4	7.4	7.6	1.6	1.0	1.4	1.3	2.7	2.3	3.6	1.6	3.9
Total Flux - %	3.2	3.8	3.9	2.8	2.9	3.2	3.1	3.3	2.4	3.5	1.8	2.2	2.5	2.8	2.8	2.8	2.8	3.0	2.8
Total silicate mineral fillers - %	63.0	60.1	58.0	58.0	55.0	62.0	62.0	62.0	62.0	59.0	64.0	47.2	38.0	58.0	58.0	58.0	58.0	61.7	58.0
Residue content after burning at 100 °C - %	62.5	60.8	60.8	58.4	58.8	64.0	59.9	61.8	61.7	61.3	63.0	58.9	55.3	59.7	59.7	61.0	58.9	59.0	58.4
Flux content as a % of residue content	5.14	6.19	6.43	4.8	4.98	4.98	5.25	5.31	3.96	5.67	2.91	3.7	4.46	4.66	4.66	4.6	4.76	5.06	4.8

Example 2

Electric cables were made using compositions B and T from the table above. Those made with composition T exhibited a high char shrinkage that resulted in cracking of the insulation layer at 1050°C, leading to insulation failure in the fire test (heating stage) according to AS/NZS 3013:1995. Cables made with the composition B that has a low char shrinkage passed the same test. The char produced was free of large visible cracks in the case of composition B whereas the char formed from composition T was heavily cracked leaving the conductor exposed.

Example 3

A composition (X) having the constituents listed in Table 2 below was prepared. Composition X was based on a commercially available ethylene propylene elastomer and silicone elastomer. The mica used was muscovite with a mean particle size of 160 μm determined by sieve analysis. Glass frit A has a softening point of 430°C and a fluxing oxide content of 30.8%. Glass frit B has a softening point of 600°C and a fluxing oxide content of 5.1%. Glass fibers A, B and C have softening points of 580°C, 650°C and 532°C, respectively and fluxing oxide contents of 12-15%. Di(t-butylperoxyisopropyl) benzene peroxide was included in the compositions for effecting thermal crosslinking. All compositions listed in this example are given in %wt/wt.

Table 2

Components	**(%wt/wt)**
Ethylene propylene rubber	27
Silicone polymer	8
Muscovite mica	20
Glass frit B	2
Clay	28
Talc	10
Zinc oxide	2

Peroxide	2
Antioxidants, coagents	1
Total	100
Total Flux (%)	2.8
Fluxing oxide as a percent of residue	4.6

Example 3.1

Specimens of Composition X for strength testing were made with dimensions 50 mm x 14 mm x 3 mm and thermally crosslinked. For comparative purposes, test specimens were similarly prepared using a commercially available silicone-based material (Composition Y), which also formed a ceramic material when heated. The samples were heated to 1000°C in air for 30 minutes and then cooled. The flexural strength of the cooled samples was determined in three point bend mode using an Instron Universal Testing Machine. Shrinkage of samples after firing was determined by measuring the lengths of the two long sides of each sample before and after firing and averaging the percentage shrinkage. The results are shown in Table 3.

Table 3

Compositions	**Flexural Strength (MPa)**	**Shrinkage (%)**
Composition X	5.9	1.6
Composition Y	4.2	4.9

The results obtained from flexural strength measurements show that Composition X has a higher flexural strength than the silicone-based composition (Y) after firing in air at 1000°C.

Shape retention is a critical factor in many applications for these types of materials, for example in electrical cable insulation. Measurements of linear shrinkage after firing at 1000°C in air showed that Composition X had superior shape retention properties in comparison to Composition Y.

Example 3.2

A 35 mm^2 compacted copper conductor was insulated with 1.2 mm wall thickness of Composition X by an extrusion process. The insulated conductor was then sheathed with a thermoplastic flame retardant halogen free material to a wall thickness of 1.4 mm. Three samples of the cable, approximately 2.5 metres long, were installed on a ladder type cable tray in an "S"configuration with bend ratios of 10 times the cable diameter. The tray was mounted on a concrete slab and used to form the top of a pilot furnace capable of following the standard temperature-time curve of the Australian Standard AS1540. Each sample cable was connected to a three phase electrical supply such that the cables were on different phases. In each circuit was a 60W light bulb and a 4A fuse. The line voltage was 240V AC. The test was started and continued for 121 minutes, at which time the temperature in the furnace was approximately 1,050°C. At the completion of this time, the circuit integrity of all of the samples was maintained. A water jet spray was then trained on the cables and circuit integrity continued to be maintained.

Example 3.3

Composition X was modified by adding small amounts of various inorganic additives in the proportions outlined in the table below. The inorganic additives included glass fibre, glass frit and alumino-silicate fibre. Composition X and the modified versions were thermally crosslinked (170°C, 30 minutes, 7 MPa) into flat sheets 2 mm thick. Samples 19 mm x 32 mm were cut out of the sheets and fired to 1000°C in air for 30 minutes. After cooling, the samples were tested for water resistance by placing a drop of water on the sample surface. The material was deemed to be water resistant if a drop of water remained on the sample surface for more than three minutes without any visual sign of absorption. The material was not considered water resistant if the water drop was completely absorbed in less than three minutes. The results of this test are shown in Table 4.

Table 4

Composition	Water Resistant
Composition X	No
Composition X/Glass fibre A (98:2)	Yes
Composition X/Glass fibre A/alumino-silicate fibre (96:2:2)	Yes
Composition X/Glass fibre B (98:2)	Yes
Composition X/Glass frit A (98:2)	Yes
Composition X/Glass frit A/alumino-silicate fibre (96:2:2)	Yes

A water drop placed on an unmodified Composition X sample was absorbed instantly. From visual inspection samples with the inorganic additives had a glassy, shiny surface layer.

Example 3.4

Six samples corresponding to the six compositions in the previous example were sectioned such that their thickness was reduced from 2 mm to 1 mm. Samples 19 mm x 32 mm were then fired to 1000°C in air for 30 minutes. After cooling they were then tested for water resistance in the same manner described in the preceding example. In all six cases the samples absorbed a drop of water placed on their surface in less than one minute, indicating a lack of water resistance. A comparison with the results in the previous example shows that sample thickness is a factor in developing water resistance.

Example 3.5

(A)

Sections of 2.7 mm^2 copper wire were insulated with Composition X and modifications to this composition as outlined in Tables 5 and 6. The wall thicknesses were set at 1.2 mm and 0.6 mm to obtain cables with thick and thin insulation layers. Insulated cables were

put together to form twisted pairs. Each twisted pair was exposed to a Bunsen burner flame for 10 minutes. The burner and cable were configured so that the peak temperature at the flame-sample interface was measured at 1020°C. The cable was allowed to cool and water was dripped across the portion of the twisted pair to assess the time taken for the circuit to short. During the burner and water test the resistance between the two wires in the twisted pair was monitored using a 500 V DC test unit. Failure in either test was deemed to be the measured resistance dropping to approximately 0 MΩ at any point in the test. The compositions and their performance in the tests for thick insulation layers are shown in Table 5 and for thin insulation layers are shown in Table 6.

Table 5

Composition	**Burner Test (pass/fail)**	**Water Test (time to short)**
Composition X	Pass	<30 seconds
Composition X/alumino-silicate fibre (99:1)	Pass	>3 minutes
Composition X/Glass frit A (99:1)	Pass	>3 minutes
Composition X/alumino-silicate fibre/Glass frit A (99:0.5:0.5)	Pass	>3 minutes

The results in Table 5 showed that additions of glass frit and/or alumino-silicate fibre in amounts totalling no more than 1% wt/wt imparted good water resistance properties to Composition X. Composition X, without any additions, had almost negligible water resistance, with a short circuit occurring less than 30 seconds after water contacted the cable.

Table 6

Composition	**Burner Test (pass/fail)**	**Water Test (time to short)**
Composition X	Pass	<30 seconds
Composition X/alumino-silicate fibre/Glass frit A (98.5:0.5:1)	Pass	<30 seconds

Composition X/alumino-silicate fibre/Glass fibre A (97:1:2)	Pass	<1 minute
Composition X/alumino-silicate fibre/Glass fibre A (96:1:3)	Pass	<30 seconds
Composition X/alumino-silicate fibre/Glass fibre A /Glass frit A (94:1:3:2)	Pass	<30 seconds
Composition X/alumino-silicate fibre/Glass fibre C (94:1:5)	Pass	<30 seconds

The results in Table 6 showed that when a change from 1.2 mm to 0.6 mm wall thicknesses was made, no tested composition exhibited acceptable water resistance. Again, this demonstrates that the thickness of the sample is a factor in developing water resistance.

(B)

In order to improve the water resistance of thin wall (0.6mm) cables, an addition of alumino silicate fibre and mica were made to Composition X to give a new composition consisting of Composition X/alumino silicate fibre/mica (94:1:5). The fluxing oxide content of the residue obtained after heating the new composition at 1000°C for 30 minutes was 5.1%. The composition was formed into thin wall twisted pair cables and tested in the burner according to the procedure described in the previous example. The wire passed the burner test and the time to short in the water test was greater than 3 minutes.

Example 3.6

Fired 2 mm thick samples of Composition X modified with inorganic additives to improve water resistance were analysed by scanning electron microscopy in order to assess the reason for their water resistance. Micrographs of the sample cross-section showed that a dense glassy layer ranging up to 10 μm in thickness was present at the surface. This glassy film overlays the porous bulk of the material, protecting it from water absorption.

Example 4

A composition with an EPDM polymer(20%), talc (30%), muscovite mica (29%) and processing aids and stabilisers was prepared on a two roll mill. At the completion of mixing, it was separated into two equal portions. One portion was returned to the two roll mill, and 2% of dicumyl peroxide was added. The two portions were then placed separately into picture frame moulds and pressed at 1,000 kPa and 170 °C for 30 minutes. At the end of this period, the press was cooled while maintaining pressure, and after the temperature had reduced to 50 °C, the pressure was reduced and the samples removed. The end result was two sheets of material that had undergone the same heat history, but one had been crosslinked while the other was thermoplastic.

Samples of dimensions 38mm x 13mm were cut from the sheets, and the dimensions accurately recorded. The samples were then placed into a muffle furnace that had been preheated to 1,000 °C. After 30 minutes exposure, the samples were removed from the furnace and allowed to cool to ambient temperature. The dimensions were then accurately re-measured and the shrinkage calculated.

It was found that the thermoplastic version showed less surface disruption than the crosslinked version, expanded less in thickness, but slightly more in length and width. This illustrates that crosslinking of the composition is not essential to achieving an acceptable performance in net shape retention after exposure to 1,000 °C.

Throughout this specification and the claims which follow, unless the context requires otherwise, the word "comprise", and variations such as "comprises" and "comprising", will be understood to imply the inclusion of a stated integer or step or group of integers or steps but not the exclusion of any other integer or step or group of integers or steps.

Dated this 30th day of June 2003
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